Tribology and Mechanical Behavior of Coatings and Engineered Surfaces
Room Grand Hall - Session EP-ThP

Tribology and Mechanical Behavior of Coatings and Engineered Surfaces (Symposium E) Poster Session

EP-ThP-2 Deposition of DLC/Si-N Composite Films Synthesized by Sputtering-PBII Hybrid System and Their Thermal Stability*, **Anas Melih**, K Yamada, S Watanabe,* Nippon Institute of Technology, Japan

Diamond-like carbon (DLC) is a metastable amorphous film that exhibits unique properties. However, many limitations exist regarding the use of DLC, for example, its tribological characteristics at high temperature, as well as its limited thermal stability. In this study, silicon/nitrogen incorporated diamond-like carbon (DLC/Si-N) composite films are studied, taking into account the thermal stability and tribological performance of these films compared with pure DLC. All the films were synthesized using RF magnetron sputtering combination with PBII techniques (so-called Sputtering-PBII Hybrid System). High purity of Silicon nitride (99.9%) disk was used as a target with RF power of 500-700 W. The substrates were also applied with negative-pulsed bias voltage of 5 kV. The mixtures of Ar-CH_4 were used as reactive gas by varying CH_4 partial pressure between 0 and 0.15 Pa, while total gas pressure and total gas flow were fixed at 0.30 Pa and 30 sccm, respectively. The structure of the films was characterized using Raman spectroscopy. The thermal stability of the films was measured using thermogravimetric and differential thermal analysis (TG-DTA). The friction coefficient of the films was assessed using ball-on-disk friction testing. The results indicate that DLC/Si-N composite films present better thermal stability due to the presence of Si-O networks in the films. The DLC/Si-N (about 11 at.%Si, 18.5 at.%N) film was showed good thermal stability in an air atmosphere with increasing temperature until 800°C. Further, the films exhibit excellent tribological performance at 500°C in an air atmosphere. It is concluded that the DLC/Si-N composite films improve upon the thermal stability and tribological performance of DLC at a high temperature .

EP-ThP-3 Mechanical and Tribological Performance of TiAlN, TaN and Nanolayered TiAlN/TaN Coatings Deposited by DC Magnetron Sputtering*, **Elbert Contreras**, J Cortínez, M Gómez,* Universidad de Antioquia, Colombia*; A Hurtado,* Centro de Investigación en Materiales Avanzados CIMAV, Mexico

Nanoscale multilayer systems have become the most important way to increase the mechanical and tribological performance of wear protective coatings. In the present research, nanoscale TiAlN/TaN multilayer coatings were deposited by DC magnetron sputtering, controlling the substrate rotation speed in order to obtain different periods and to evaluate their effect on mechanical and tribological properties; additionally, monolayer TiAlN and TaN coatings were deposited to compare the effect of multilayer architecture on the coatings performance. SEM images showed a well define columnar and homogenous microstructure, it was possible to observed the multilayer architecture with higer bilayer periods. TEM images exhibited well defined interface and diffusion between both TiAlN and TaN layers. A high crystalline coatings, change in the growth direction of coatings and residual stresses was the most relevant results obtained by XRD. Regarding to the residual stresses, it was observed that monolayer coatings exhibit high compressive stresses reaching values up to 8 GPa. On the other hand, the residual stresses of nanoscale multilayer coatings exhibit considerable lower values and decrease progressively as the bilayer period of the coatings decreases, reaching values below 2 GPa. TiAlN and TaN coatings exhibited hardness of 16 GPa and 17 GPa, respectively, while TiAlN/TaN coatings showed a progressive increase as the coating period decreases, reaching values up to 22 GPa with bilayer periods lower than 10 nm. Scratch test results showed that all nanoscale multilayer coatings exhibit better adherence compared to TiAlN and TaN monolayer coatings. Additionally, it was also observed a slight tendency to increase the adherence once the bilayer period decreases reaching values up to Lc = 42 N. Fracture toughness was evaluated by nanoindentation, it was possible to calculate a significant improvement in the toughness of multilayer coatings and a significant increase in the H^3/E^2 ratio. With respect to the tribological performance, it was possible to observe a decrease in friction coefficient and wear rates of nanoscale multilayer coatings compare to TiAlN and TaN monolayer coatings. Using cross-section FIB it was possible to observe initial deformation in the constituent layers followed by the propagation of cracks along the interfaces between TiAlN and TaN layers, in nanoindentation tests, scratch tests and tribological tracks.

EP-ThP-4 Development of Catalytically Active Nano-Composite Coating for Severe Boundary Lubricated Conditions of Hydraulic Fluids*, V DaSilva, **Osman Levent Eryilmaz**, A Erdemir,* Argonne National Laboratory, USA

Applying thin coatings onto bulk materials is a surface benefactor favorable in tribology because of its ability to lower friction and copiously improve wear performance. These phenomena result in more reliable mechanical systems and improve energy efficiency. In this work, Vanadium Nitride - Copper coatings were produced and optimized (power of each target was adjusted to achieve a range of Cu concentration within the hard nitride phase) using HIPIMS magnetron sputtering system. The thin coatings were deposited onto 52100 steel flat, cylinders, and ball samples for tribological testing. Characterization such as surface roughness, composition, coating thickness, and adhesion were analyzed using a wide range of characterization techniques prior to testing. The coatings were evaluated in hydraulic fluids under unidirectional sliding (ball on flat) using two different test configurations. The results showed that, use of coating on both static and moving surfaces significantly improved wear resistance and lowered the coefficient of friction when tested under severe conditions (1 GPa maximum Hertzian contact pressure). Post-test microscopy and profilometry analysis confirmed ultralow wear on sliding surfaces. Furthermore, the VN-Cu coated surfaces showed an astounding 30-40% reduction in coefficient of friction compared to baseline 52100 ball on 52100 flat in hydraulic fluid.

EP-ThP-5 Size-Independent High Strength of CuTi/Ti Metallic Glass/Crystalline Nanolayers*, M Abboud,* Middle East Technical University, Turkey*; A Motallebzadeh,* Koç University, Turkey*; **Sezer Özerinç**,* Middle East Technical University, Turkey

Thin film metallic glasses (MG) offer a wide design space for the development of hard, corrosion resistant and biocompatible coatings. However, one of the major disadvantages of MGs is their brittle nature. Utilizing a nanolayered MG – crystalline structure is a promising approach for improving the ductility of these materials. Hardness of these nanocomposites usually increases with decreasing layer thickness, and it is lower than that of the corresponding monolithic MG. In this work, we demonstrated a nanolayered model system composed of CuTi MG and nanocrystalline Ti layers (CuTi/Ti) that shows unusually high hardness, comparable to that of monolithic CuTi MG. Furthermore, we did not observe any significant dependence of strength on layer thickness.

We prepared thin films of nanocrystalline Ti, CuTi MG, and nanolayered CuTi/Ti. A magnetron sputterer deposited 1 µm-thick films on oxidized silicon substrates, and layer thickness was varied in the range 10–100 nm. X-ray diffraction and electron microscopy verified the microstructure with alternating layers, and nanoindentation experiments measured the mechanical properties.

Nanocrystalline Ti sample has an average grain size of ≈20 nm and the grain size of Ti layers in the CuTi/Ti samples varies in the range 5–16 nm; monotonically increasing with increasing layer thickness. Hardness of all CuTi/Ti nanolayers are in the range 7.1–7.3 ±0.3 GPa with no observable layer thickness dependence; and are comparable to that of monolithic CuTi MG (7.0 ±0.3 GPa). On the other hand, 1 µm-thick nanocrystalline Ti has a hardness of only 0.9 GPa. An order of magnitude higher hardness of the nanocomposite when compared to the crystalline Ti suggests that the confinement of hexagonal close-packed Ti in between amorphous layers results in unexpected levels of extrinsic strengthening. In order to have a comparison, we also prepared nanolayered CuTi MG/crystalline Cu samples. These layers are softer than CuTi for all layer thicknesses, and show the usual size dependence of increasing hardness with decreasing layer thickness, in the range 5.1–6.3 GPa. These differences between the samples containing HCP Ti layers and FCC Cu layers suggest that crystal structure plays an important role in the mechanical behavior of nanolayered MG-crystalline composites.

The results demonstrate that by the appropriate selection of the constituent layers, it is possible to design size-independent high strength nanolayered metals. This enables the opportunity of engineering the layer thicknesses for optimizing other properties of nanolayers such as ductility, corrosion resistance and biocompatibility for application requirements.

EP-ThP-6 Extended Crack-free Tensile Deformation of Ultrathin Metallic Glass Films Due to an Intrinsic Size Effect*, Oleksandr Glushko,* Erich Schmid Institute of Materials Science, Austria*; M Mühlbacher,* Montanuniversität Leoben, Austria*; C Gammer, M Cordill,* Erich Schmid Institute of Materials Science, Austria*; C Mitterer,* Montanuniversität Leoben, Austria*; J Eckert,* Erich Schmid Institute of Materials Science, Austria

Although metallic glasses (MGs) exhibit a unique combination of mechanical and chemical properties (high strength, hardness, elastic strain, wear and corrosion resistance, biocompatibility), their application as structural or functional materials is hindered by the lack of ductility which leads to catastrophic brittle-like fracture. When the size of a MG sample is reduced below some critical value, typically of the order of a few hundred nanometers, then considerable ductility can be observed. However, this size effect was demonstrated so far mostly by nanomechanical testing inside a transmission electron microscope using samples prepared by focused ion beam (FIB) milling. Whether the ductile-like behavior of submicrometer-sized metallic glasses is a real "intrinsic" size effect or it is rather caused by extrinsic factors like sample shape, ion beam effect or parameters of the testing setup is currently a subject of extensive discussions in the community.

In this contribution the tensile properties of thin film PdSi MGs grown by sputter deposition on a polymer substrate are considered. The integrity of the MG films during stretching was monitored by in-situ measurements of the electrical resistance. Although the 250 nm thick films fail in a brittle manner at 2% strain, considerable local ductility originating from the substrate constraint is observed. A strong size effect on the deformation behavior appears when the film thickness drops below 16 nm. The 7 nm thick films with the same composition show a crack-free deformation up to a strain of 7%. Even at higher strains no brittle-like failure but rather short and isolated cracks are observed. Cyclic tensile loading revealed extreme fracture resistance of ultrathin amorphous films showing no cracks after 30000 stretching cycles with a strain amplitude of 2%. Since all tests are performed at ambient conditions on films deposited using an industrially scalable process, the demonstrated size effect can be directly utilized for applications, such as protective coatings, nanoelectromechanical devices or half-transparent conductive layers for flexible electronics.

EP-ThP-11 Effect of Surface Treatments on AISI H13 Steels*, Marco Antonio Doñu Ruiz,* Universidad Politecnica del Valle de México, Mexico, México*; M Buenrostro Arvizu,* Universidad Autonoma Metropolitana Azcapotzalco, Mexico*; N Lopez Perrusquia,* Universidad Politecnica del Valle de México, Mexico, México*; V Cortés Suárez,* Universidad Autonoma Metropolitana Azcapotzalco, Mexico*; C Torres San Miguel,* Instituto Politecnico Nacional, Mexico*; G Pérez Mendoza,* Universidad Politecnica del Valle de Mexico

Mechanical properties can be improved by thermochemical treatments, the present work studied the effect of surface treatment on AISI H13. Nitriding was performed according to TENIFER® process under two conditions; i) samples of were employed in the salt bath component at temperature of 853 K with exposure time of 1h 3min ii) sample were austenized in a vacuum furnace at a temperature of 1283 K for 90 minutes and cooled with nitrogen to 333 K. After all the samples were tempered, the first was carried out at 813 K and the second at 878 K, with 2 h, respectively. After hardening and tempering (HT) , the samples HT were nitride (HTN).

Boro nitride coating was applied on samples on AISI H13 in two stage: i) samples were boride by paste dehydrated pack at temperatures 1173 K for 3 hours and then nitriding on sample boride at temperature of 853 K with exposure time of 1h 30 min.

The microstructural and phases composition were characterized using scanning electron microscopy (SEM) and X-ray diffractometer (XRD). The samples were mechanical characterization by three-point bend test and microhadness test.

Nitrided coating thicknesses are over thicknesses of 4.97 um with zone of diffusion of 52.8 um of thickness, for the case of boronitride a depth of 26.42 um is observed. For the condition AISI H13 hardening, tempering and nitriding a maximum bending stress of 10990 MPa was obtained in comparison with the AISI H13 as received with a value of 3677 MPa.

EP-ThP-12 Mechanical Properties and Fretting Corrosion of Zr/ZrN/CNx Hierarchical Multilayers Deposited by HIPIMS on Ti Biomedical Alloy*, Martin Flores,* *J Perez, O Jiménez, L Flores,* Universidad de Guadalajara, Mexico

The hierarchical multilayers of hard ceramic coatings can improve the adhesion to substrates and the tribocorrosion resistance of biomedical alloy as Ti6Al4V. The coatings deposited by HIPIMS have a more compact growth respect to films deposited by DC magnetron sputtering; this diminish the presence of pinholes and defects that permit to predict a reduction in permeability. The design of the multilayer was made with a top layer of CNx in order to reduce the friction coefficient. In this work we study the mechanical and fretting corrosion behaviour of Ti6Al4V biomedical alloys coated with multilayers of Zr/ZrN/CNx and Zr/ZrN deposited by HIPIMS (High Power Impulse Magnetron Sputtering). The fretting corrosion tests were made in fetal bovine serum (FBS) to simulate the proteins in synovial liquid. The tribocorrosion was studied using open circuit potential (OCP) and potentiostatic polarization in the passive region during the relative movement of samples. The friction coefficient was measured as a function of the applied potential. The worn surface at wear track was analysed by SEM, XRD and Raman spectroscopy. We report the results on composition and structure of tribolayer and removed material of the wear tracks, and the comparative study in mechanical properties fretting corrosion and friction behaviour of coatings of Zr/ZrN and Zr/ZrN with a top layer of CNx.

EP-ThP-13 Quantum Tools for Life-Time Prediction of Coatings and Thin Films*, Norbert Schwarzer,* SIO, Germany

Everybody who ever tried to do some life-time prediction on parts subjected to erosion or tribology or any other form of complex and potentially multi-body interaction full well understands and values Mark Twain's famous dictum about the simple fact that making predictions is very complicated… especially if they concern the future. The smaller the objects and the more complex their internal structure the even more complicated this task becomes. In thin-film physics the most principle uncertainties come on top of the already rather nonlinear and often chaotic behavior the objects of interest are subjected to.

Instead of seeing this increase of complexity due to the incorporation of roughness, defects, impurity and quantum effects as an additional problem in our efforts to come to better life-time predictions with respect to all sorts of coating-applications, this author actually saw an interesting potential in the apparently tedious "add-on-effects" coming into play with smaller and smaller scales.

By incorporating quantum theoretical concepts into highly sophisticated physical models [1] we obtained a very compact, rather general and powerful tool to handle practical applications in many different fields, leading to a much more holistic uncertainty budget calculation and – as a surprise – even a more comfortable and secure life-time prediction.

[1] N. Schwarzer, "The Theory of Everything - Quantum and Relativity is everywhere – A Fermat Universe", Pan Stanford Publishing, July 2018, ISBN-10: 9814774472

EP-ThP-16 Structure and Fretting Wear Behavior of CuNiIn/MoS_2-Ti Multilayers Fabricated by Magnetron Sputtering Method*, Chunbei Wei,* *Q Li, S Lin, H Hou, M Dai,* Guangdong Institute of New Materials, China

Fretting denotes a tribological phenomenon related to movements of small amplitudes between two surfaces in contact. This working condition occurs in many mechanical assemblies where the parts are occasionally subjected to a vibrating environment or are sustaining variable stresses directly. It often results in damage that may lead to premature component failure. A thick soft CuNiIn coating prepared by plasma sprayed is usually used to cover engine components from fretting due to its great capacity of accommodation by plastic deformation. However, CuNiIn coating has limited life because of its high friction coefficient. Some researches combine CuniIn+polymer bonded lubricant MoS_2 to improve the tribological behavior due to the cohesion, ductility and adhesion of third bodies inside the contact.

In this paper, CuNiIn/MoS_2-Ti multilayer was fabricated by magnetron sputtering method. As a comparison, CuNiIn monolayer and MoS_2-Ti monolayer was also prepared. The linear fretting wear was used to study the fretting wear behavior of the coatings. The results show that the interface of multilayer prevented the growth of thick columnar crystal of CuNiIn interlayer and MoS_2-Ti interlayer, whereas the monolayer showed thick columnar structure. A compact, fine crystal structure has formed for CuNiIn/MoS_2-Ti multilayers. The microhardness of the multilayers was in the range of 350~500HV, which was higher compared with that of the CuNiIn and MoS_2-Ti monolayer. After 12000 cycles of fretting wear, the friction coefficient of CuNiIn monolayer was more than 1.0, and its wear trace depth reached to 108μm, indicated the failure of the coating. However, the friction coefficient of MoS_2-Ti monolayer was about 0.1, and the wear trace depth was 4.5μm. MoS_2-Ti monolayer exhibited good lubricant properties, leading to the improvement of the tribological performance. The friction coefficient of CuNiIn/MoS_2-Ti multilayer was lower to 0.07, and the wear trace depth was 0.5 μm, demonstrated that the better wear properties of the multilayer compared with that of the monolayer. These can attribute to the more compact structure and higher ductile of the multilayers, as well as its good lubricant characteristic.

EP-ThP-17 Contact-focusing Electron Flow (CFEF) Induced Near-zero Running-in for Low Friction of Carbon-steel contact Interface*, Dongfeng Diao,* Institute of Nanosurface Science and Engineering, Shenzhen University, China

Under air condition, a-C film sliding against steel surface often shows high friction coefficients (μ=0.1~0.3), which largely impedes its potential in industrially. In this study, we report a new method of contact-focusing electron flow (CFEF) by applying direct current (DC) at the sliding interface between a-C film and steel ball to trigger the in-situ formation of nanosized graphene sheets, and induce a fast low-friction behavior. The developments of the friction behavior with different DC values and normal loads were examined. The typical friction coefficient dropped one order of magnitude from 0.2 to 0.02 and the run-in period was close to zero under CFEF-condition. The low friction interface, especially the carbon-transfer film was examine at nanoscale by using the newest detecting avenues. The nanostructure of the transfer films was observed by Cs-corrected TEM and EELS analysis. The elemental composition of the transfer film was investigated by using EDS, XPS and Raman spectra analysis. Based on these analyses, we found three key factors for the fast low friction (near-zero running-in). (1) CFEF excited the carbon structural transition from amorphous to graphene sheet at the contact area. (2) Friction transferred the nanosized graphene sheets to the steel ball surface. (3) CFEF-friction coupling effect supplied the nanosized graphene sheets into the sliding interface. This work shed light on the CFEF-method for the in-situ fabrication of nanosized graphene sheets at the carbon-steel contact interface for low friction with near-zero running-in period.

EP-ThP-18 Misinterpreting Size-effects during Coating Nanoindentation*, Esteban Broitman,* SKF Research & Technology Development Center, Netherlands

The hardness of a material is a multifunctional physical property depending on a large number of internal and external factors. The transition from macroscale to microscale, and from microscale to nanoscale indentation hardness measurement is accompanied by a decreasing influence of some of these factors and by an increasing contribution from others.

The Vickers indenter used in macro-indentation tests has been designed to give geometrically similar indentations (i.e. the strain generated within the material is independent of the load), so the hardness should be independent of the indentation size. This fact results to be true for macroscale indentations. However, for microscale indentations (loads of less than 1N), it is well established that the hardness decreases or, more frequently, increases with the decrease of the applied load. This effect is known as the *"indentation size effect"* (ISE). The same phenomena has also been observed during coating nanoindentations at very low loads (low indentation depths), using either Berkovich or cube-corner indenters.

The gradient plasticity theory attributes ISE to the evolution of the so-called geometrically necessary dislocations beneath the indenter, which gives rise to strain gradients that affect the measurements at low penetration depths. In this presentation we will show that most of the ISE reported in the literature are, in fact, related to artifacts originated from a wrong surface preparation that generates work hardening, or the lack of surface microstructural characterization, which leads to ignore the presence of surface roughness, grain precipitates, or very thin surface oxide layers. Furthermore, we will show also that ignoring pile-ups or the use of an incorrect indenter geometric calibration generate false ISE results. We conclude that most of reported ISE have been misinterpreted and can be suppressed when proper data interpretation and processing are used.

EP-ThP-19 e-Poster Presentation: Think You Have Produced DLC? Think Again!*, Arman Mohammad Khan, H Wu, Y Chung, Q Wang,* Northwestern University, USA

Carbon-containing tribofilms have been widely reported to form in lubricated tribological contacts. These films show the same Raman *D* and *G* signatures as those of graphite- or diamond-like carbon (DLC) films. As a result, these films are commonly assumed to be DLC. Our work has revealed that this interpretation may not be always correct. In our experiments, we formed such tribofilms in standard pin-on-disk tribotesting using steel counter-faces and polyalphaolefin (PAO) base oil with cyclopropanecarboxylic acid (CPCa) as an additive. Raman spectra obtained from these tribofilms have *D* and *G* features identical to those from DLC. Multiple analytical techniques (micro-FTIR, mass spectrometry, and proton NMR) coupled with reactive molecular dynamics simulations demonstrate that these tribofilms are not DLC, but are in fact high molecular weight hydrocarbons acting as a solid lubricant.

EP-ThP-20 Mechanical and Tribological Properties of Cr-Al-Si-N-O Coatings Prepared by Arc Ion Plating for Cutting Tools*, Jun-Ho Kim, W Kim,* Korea Institute of Industrial Technology (KITECH), Republic of Korea

Quinary Cr-Al-Si-N-O nanocomposite films were deposited onto WC-Co substrate by a filtered arc ion plating system using $CrAl_2$ and Cr_4Si composite targets under N_2/Ar atmosphere. XRD and XPS analyses revealed that the synthesized Cr-Al-Si-N-O films were nanocomposite consisting of nanosized (Cr,Al,Si)N crystallites embedded in an amorphous Si_3N_4/SiO_2 matrix. The hardness of the Cr-Al-Si-N-O films exhibited the maximum hardness values of ~40 GPa at a Si content of ~2.78 at.% due to the microstructural change to a nanocomposite as well as the solid-solution hardening. Besides, Cr-Al-Si-N-O film with Si content of around 2.78 at.% also showed perfect adhesive strength value of 109 N. These excellent mechanical properties of Cr-Al-Si-N-O films could be help to improve the performance of machining tools and cutting tools with application of the film. Also X-ray diffractometer (XRD) analysis was conducted to investigate the crystallinity and phase transformation of the films. Moreover, it was found that the improved nanohardness and the H/E ratio contributed to excellent wear resistance of the coatings. The friction coefficient and wear rate of the Cr-Al-Si-N-O coatings first decreased and then increased with increasing silicon content. The friction coefficient and wear rate were also mainly related to the lubricant wear debris in this work.

EP-ThP-22 Friction Property of Si-DLC with Scratch Damage Before and After Local Repairing Deposition to the Scratch Scar*, H Takamatsu, K Tanaka,* ***Akihiko Ito****, H Kousaka, T Furuki,* Gifu University, Japan

Diamond-Like Carbon (DLC) has excellent mechanical properties such as high hardness (over 10GPa), high wear resistance, and low friction. However, DLC film has low impact resistance and toughness because of its high hardness and brittleness, and thus it is often damaged by cracking from film surface and peeling from base material.

DLC film with local damages is repaired through the following process: (1) Removal of all the DLC film, (2) Recoating of new DLC film. However, this process is not efficient. We have proposed a new way of repairing, or recoating only to local damaged points of DLC by using a micro coating device. In this paper, we discuss the friction property of Si-DLC on which a local damaged point is repaired by recoating.

A local damage was introduced to Si-DLC pre-coated steel disk (SUJ2, JIS) by scratching at an almost constant load. Recoating of Si-DLC was conducted to the Si-DLC disk with a slit mask so that only the area near the scratch is recoated.

In order to evaluate the friction behavior of pre-coated, damaged and repaired Si-DLC films, these films were slid against a steel ball (SUJ2, JIS) at a vertical load of 15N and a rotation speed of 10m/min by ball on disk friction tester.

During running-in process, damaged Si-DLC showed frequent fluctuation of friction coefficient, which appeared from the sliding distance of about 60m. After the process has done, large increase of friction coefficient was sometimes confirmed with passing through the scratch scar. On the other hand, such fluctuation and unstable transition of friction coefficient were not confirmed in the repaired Si-DLC.

Transfer film formed onto the mating steel ball surface is considered to easily re-adhere to the metal exposed part in the damaged Si-DLC. Such transfer film detached from the ball surface can easily stick back to the ball surface, because of initial oxide film on the ball which prevents the formation of transfer film is already removed during the initial stage of sliding. We assume the fluctuation of friction coefficient in the damaged Si-

DLC was caused by such detachment and re-attachment of transfer film at the sliding interface. In addition, we consider that such detachment of transfer film was suppressed in the repaired Si-DLC because the metal exposed part is covered by recoating of Si-DLC. As result, it is possible that partially repairing local damaged DLC shows better friction properties.

EP-ThP-23 Raman Scattering Characterizes Thermally Annealed HiPIMS Sputtered MoS2 Coatings*, **Henning Moldenhauer**, W Tillmann, A Wittig, D Kokalj, D Stangier, A Brümmer, J Debus,* TU Dortmund University, Germany

Omnipresent friction forces and wear limit the service time of machine parts. The usage of lubricants increases the service time remarkably due to a reduction in the wear. In that context, solid lubricants, like MoS_2 coatings grown by physical vapor deposition (PVD), are highly promising. However, the friction coefficient and, consequently, the lifetime of the coating depend on its oxidation resistance which in turn depends on several microscopic parameters, like its structure. In order to produce highly oxidation resistant coatings, high-power impulse magnetron sputtering (HiPIMS) is used. Their structural properties at the nanoscale are characterized by laser-light scattering methods; in particular, Raman scattering studies on HiPIMS sputtered MoS_2 thin films are performed aiming at their structural and tribological properties under multiple annealing conditions.

Raman scattering spectroscopy is a non-destructive and contactless tool for identifying the structure, morphology and chemistry of material surfaces. The material's quantized lattice vibrations are laser-excited and optically measured, thus generating a physicochemical fingerprint of the surface at the atomic scale. Raman scattering spectroscopy provides a high spatial resolution of about one micrometer and can be used for practically any sample geometry.

Using this technique, a microscopic layering of MoS_2 is detected: Interlayer-phonon modes at low frequency (38 cm^{-1}) build up for increasing annealing temperatures of the coatings (until 600°C). Changes in the intensity ratio of the in-plane and out-of-plane Raman modes moreover hint at crystal symmetry changes. To assess the tribological performance, ball-on-disc experiments are performed in combination with Raman scattering. A dependence of the friction coefficient on the annealing temperature and the layering of the MoS_2 is found. Furthermore, the MoS_2 typical needle-like structure and a denser morphology of the MoS_2 thin films compared to a DC sputtered reference are revealed by scanning-electron microscopy. A hexagonal crystallization structure and the crystallite size are evaluated from X-ray diffraction.

EP-ThP-24 Friction Reduction in Sliding Between Si-DLC vs. Steel Ball by Ar Plasma Irradiation Using Microwave-excited Atmospheric Pressure Plasma Jet*, T Hibino,* ***Hiroyuki Kousaka****, T Furuki,* Gifu University, Japan*; J Kim, H Sakakita,* National Institute of Advanced Industrial Science and Technology (AIST), Japan

Diamond-like carbon (DLC) has widespread applications in many fields due to its excellent mechanical properties such as high hardness, low friction, chemical inertness, and so on. Recently, DLC is applied to machine parts as surface coating to improve frictional property. The interface between DLC and mating surface shows low friction through running-in period, in which shear strength at the interface decreases gradually; in other words, the interface between as-deposit DLC and mating surface does not show low-friction from the very beginning of sliding. Since high-friction during running-in period may cause severe damage to sliding interface such as the fracture of DLC coating, shortening of running-in period is desired. In our previous work, Okumura et al. investigated the influence of atmospheric He plasma irradiation to the sliding interface between a polyacetal ball and Si-doped DLC disk, demonstrating that the running-in period is shortened by plasma irradiation [1]. In this work, we further tried to use plasma irradiation for shortening the running-in period in the sliding between a steel ball and Si-doped DLC disk. A Si-doped DLC (a-C:H:Si) with a Si content of 27 at % was coated by using DC plasma enhanced chemical vapor deposition (PECVD) onto a steel disk (SUS304, JIS) 25 mm in diameter. The Si-DLC disk was slid against a stainless-steel ball (SUS440C, JIS) 8mm in diameter under dry condition at a normal load of 1 N and rotation speed of 0.67 m/s for 1800 m. During the initial sliding distance of 100 m, atmospheric Ar plasma was irradiated to the sliding track on the Si-DLC disk by using microwave-excited atmospheric pressure plasma jet [2]. In the case of plasma irradiation, friction coefficient decreased to 0.1 rapidly just after stopping the plasma irradiation and it was kept from 100 to 1800 m, or the end of sliding test; on the other hand, friction coefficient without plasma irradiation was always higher than 0.1 and fluctuated more largely from 100 m to 1800 m. It was shown in the sliding between a steel ball and Si-doped DLC disk that the plasma irradiation shortened the running-in period and decreased friction coefficient. (The authors gratefully acknowledge the funding by JST CREST, Japan.)

[1]. S. Okumura, et. al, Effect of plasma irradiation on the interface between DLC and plastic, ISPlasma2017 (2017), Paper No.04P80.

[2]. J. Kim, et. al, Microwave-excited atmospheric pressure plasma jet with wide aperture for the synthesis of carbon nanomaterials, Japanese Journal of Applied Physics 54, 01AA02 (2015).

EP-ThP-27 Taguchi Method to Study Effects of Plasma Surface Texturing on Friction Reduction of Cast Iron at High Speed Sliding Lubricated Conditions*, W Zha, C Zhao, R Cai,* ***Xueyuan Nie,*** University of Windsor, Canada

Cathodic plasma electrolysis (CPE) is used to create surface texture on cast iron for the purpose of friction reduction. Micro craters formed by the explosion of gas bubbles can generate hydrodynamic force that separate two sliding surface, increase the oil film thickness and thus, decrease the friction. The Taguchi method is used to find the optimal process parameters (voltage and roughness) for surface texturing. The orthogonal array and the signal-to-noise ratio are employed to study the effect of each process parameter on friction. The results show that with higher voltage and lower roughness, the friction coefficient can be reduced to a lower value.

EP-ThP-30 Test Rig Development For Static Friction Assessment At High Temperature*, M Azzi,* Lebanese University (UL), Lebanon*; E Bitar-Nehme,* Tricomat, Canada*; J Schmitt, T Schmitt, L Martinu,* ***Jolanta-Ewa Klemberg-Sapieha,*** École Polytechnique de Montréal, Canada

The oil and gas industry uses steam-turbine power generation valves are vital equipment components that are used to control the flow of steam. Sealing surfaces in the valve must withstand high contact and friction stresses at high temperature. The main cause of valve failure is the degradation of sealing surfaces that can result in valves not cycling on demand due to high static friction. Development of materials or coatings with high tribological performance (high wear resistance and low friction) at high temperatures is the key for manufacturing valves with long service life.

In the present work, a novel experimental test rig has been developed to rigorously investigate the evolution of static friction in a tribological contact at high temperature in terms of holding time and contact stress. Pin-on-Flat configuration has been adopted in the design with the flat sample being mounted in a furnace that heats up to 800°C. The furnace moves vertically on a well-guided structure to bring the sample into contact with its counterface that moves in a linear motion generating friction.

Here, we present an approach to assess static friction, and we illustrate it by the results for stainless steel 304 and for Alumina counterface. The measurements showed that static friction increases with the number of passes to reach a plateau at 0.7 after a certain number of passes that depends on the contact stress. In addition, friction was shown to strongly depend on temperature and more importantly on the holding time at high temperature which might be related to processes such as diffusion that take place at the interface.

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